

APPLICANT: Shoichi OSADA et al.

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FOR: Semiconductor Encapsulating Epoxy Resin

Composition and Semiconductor Device

GROUP: 1712

EXAMINER: ZIMMER, MARCS

#### DECLARATION

Honorable Commissioner of Patents and Trademarks Washington, D.C. 20231

Sir,

- I, Eiichi ASANO, resident of c/o
  Silicone-Electronics Materials Research Center,
  Shin-Etsu Chemical Co., Ltd., 1-10, Oaza Hitomi,
  Matsuida-machi, Usui-gun, Gunma-ken, Japan do hereby
  declare that:
- 1. I was graduated from Faculty of High Molecular Weight Materials of Science Department of Hokkaido

University, Japan in March 1985. Since November 1985, I have been employed by Shin-Etsu Chemical Co., Ltd., the assignee of the above-identified application. I have been engaged in research and development relating to epoxy resin encapsulators in the laboratory of the Company.

- 2. I am one of the named inventors of the above-identified application and hence, am familiar with the subject matter disclosed in said application.
- 3. In order to show the feature of the present invention, I conducted the following experiments.

## [Experiment]

Epoxy resin compositions for semiconductor encapsulation were prepared by uniformly melt mixing the components shown in Table I in a hot twin-roll mill, followed by cooling and grinding. The measured properties of the compositions are shown in Table I.

The raw materials used are shown below.

#### Epoxy resins:

- (a) o-cresol novolac epoxy resin: EOCN1020-55 by Nippon Kayaku K.K. (epoxy equivalent 200)
  Curing agents:
  - (c) phenolic novolac resin: DL-92 by Meiwa Kasei
    K.K. (phenolic equivalent 110)

Zinc molybdate on inorganic carrier (zinc molybdate content 18 wt%):

KEMGARD 1261 (carrier: spherical silica with a mean particle size 0.5  $\mu m$  and a specific surface area 5.5  $m^2/g$ ) by Sherwin-Williams Co.

## Inorganic filler:

Spherical fused silica (mean particle size 20  $\mu m$ ) by Tatsumori K.K.

## Polyorganosiloxane A:

KF96 (dimethylsilicone fluid (oil), viscosity

1,000 cs at 25°C) by Shin-Etsu Chemical Co., Ltd.

Polyorganosiloxane C:

KF54 (methylphenylsilicone fluid (oil), viscosity
400 cs at 25°C) by Shin-Etsu Chemical Co., Ltd.
Polyorganosiloxane E:

XF-40 (alcohol-modified silicone oil) by Toshiba Silicone Co., Ltd.

Polyorganosiloxane F:

TSF-410 (higher fatty acid-modified silicone oil) by Toshiba Silicone Co., Ltd.

Polyorganosiloxane G:

XF-42 (epoxy-modified silicone oil) by Toshiba Silicone Co., Ltd.

Silicone rubber particles:

TREFIL E-500® (spherical silicone rubber particles having a mean particle size of 3  $\mu m$ ) by Toray-Dow Corning Silicone Co., Ltd.

Epoxy-modified silicone resin:

The same one as in Examples 9 to 16 of the present specification.

Curing accelerator:

triphenyl phosphine by Hokko Chemical K.K.

Parting agent:

Carnauba wax by Nikko Fine Products K.K.

Silane coupling agent:

KBM403 ( $\gamma$ -glycidoxypropyltrimethoxysilane) by Shin-Etsu Chemical Co., Ltd.

Properties of epoxy resin compositions were measured by the following methods.

## (1) Spiral Flow:

Measured by molding at 175°C and 70 kgf/cm<sup>2</sup> for a molding time of 90 seconds using a mold in accordance with EMMI standards.

#### (2) Hardness When Molded:

Using the method described in JIS-K6911, a rod measuring  $10 \times 4 \times 100$  mm was molded at 175°C and  $70 \, \text{kgf/cm}^2$  for a time of 90 seconds. The hardness when hot was measured with a Barcol Impressor.

# (3) Moldability

A QFP package of  $14 \times 20 \times 3.5$  mm was molded. Using an ultrasonic flaw detector, internal voids were detected.

#### (4) Flame Retardance:

A 1/16 inch thick sheet was molded and the flame retardance of the sheet was rated in accordance with UL-94 test specifications.

#### (5) Moisture Resistance:

A silicon chip measuring  $1 \times 1$  mm on which aluminum wiring had been formed was adhesively bonded to a 14-pin dual in-line package (DIP) frame

(Alloy 42), and the aluminum electrodes on the chip surface were wire bonded to the lead frame using 30 µm gold wire. The epoxy resin composition was then molded over the chip at 175°C and 70 kgf/cm² for a time of 120 seconds, and post-cured at 180°C for 4 hours. Twenty packages thus obtained were left to stand for 500 hours and 800 hours at 140°C and 85% relative humidity while being subjected to a bias voltage of 5V DC. The number of packages in which aluminum corrosion arose was counted.

## (6) Solder cracking resistance

A flat package of  $14 \times 20 \times 2.7$  mm was molded using an epoxy resin composition. It was postcured at  $180^{\circ}$  C for 4 hours, allowed to stand in a thermostat chamber at  $85^{\circ}$  C and RH 85% for 168 hours, and immersed in a solder bath at a temperature of  $220^{\circ}$  C or  $240^{\circ}$  C for 30 seconds. The outside of the package were observed to inspect cracks.

<u>Table I</u>

| Component (parts by weight)                              |         | Example |       | Comparison |       |       |       |             |
|--|---------|---------|-------|------------|-------|-------|-------|-------------|
|  |         | 1       | 2     | 3          | 4     | 5     | 6     | Invention   |
| Epoxy resin  | (a)     | 64.52   | 64.52 | 64.52      | 64.52 | 64.52 | 64.52 | 43.52       |
| Curing agent   | (c)     | 35.48   | 35.48 | 35.48      | 35.48 | 35.48 | 35.48 | 35.48       |
| Polyorgano<br>siloxane                                   | (A)     | 4       | -     | -          | -     | _     | -     | -           |
|  | (C)     | _       | 4     | _          | -     | -     | _     | -           |
|  | (E)     | -       | -     | 4          | -     | -     | _     | -           |
|  | (F)     | -       | -     | _          | 4     | -     | -     | -           |
|  | (G)     | _       | -     | _          | _     | 4     | -     | -           |
| Silicone rubber particles                                |         | -       | -     | -          | -     | _     | 4     | -           |
| Epoxy-modified silicone resin (Amount of siloxane added) |         | -       | ı     | -          | _     | -     | _     | 25.0<br>(4) |
| Inorganic filler   |         | 500     | 500   | 500        | 500   | 500   | 500   | 500         |
| Curing accelerator                                       |         | 1.2     | 1.2   | 1.2        | 1.2   | 1.2   | 1.2   | 1.2         |
| KEMGARD1261  |         | 100     | 100   | 100        | 100   | 100   | 100   | 100         |
| Parting agent  |         | 3       | 3     | 3          | 3     | 3     | 3     | 3           |
| Carbon black   |         | 2       | 2     | 2          | 2     | 2     | 2     | 2           |
| Silane coupling agent                                    |         | 1       | 1     | 1          | 1     | 1     | 1     | 1           |
| Spiral flow (cm)   |         | 95      | 83    | 90         | 74    | 81    | 70    | 82          |
| Hardness when molded                                     |         | 80      | 78    | 77         | 68    | 76    | 80    | 80          |
| Moldability  |         | ОК      | OK    | OK         | NG    | ОК    | OK    | ОК          |
| Flame retardance   |         | V-0     | V-0   | V-0        | V-0   | V-1   | V-1   | V-0         |
| Moisture<br>resistance                                   | 500 hrs | 0/20    | 0/20  | 4/20       | 0/20  | 0/20  | 0/20  | 0/20        |
|  | 800 hrs | 8/20    | 5/20  | 16/20      | 9/20  | 0/20  | 0/20  | 0/20        |
| Solder<br>cracking<br>resistance                         | 220° C  | 0/5     | 0/5   | 1/5        | 5/5   | 0/5   | 0/5   | 0/5         |
|  | 240° C  | 3/5     | 3/5   | 4/5        | 5/5   | 2/5   | 0/5   | 0/5         |

Note: In the above compositions, the molar ratio of epoxy group to phenolic hydroxy group is all the same.

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Dated this 29th day of March , 2005

Exichi Jano